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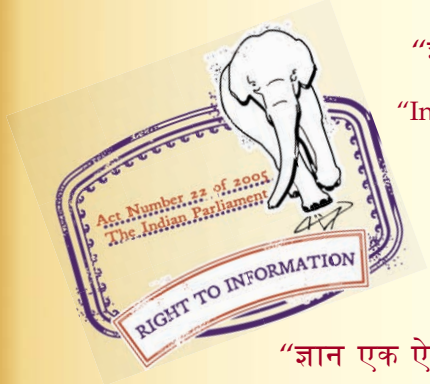

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Indian Standard
METHODS OF TEST FOR VULCANIZED RUBBERS

PART XIX PERMEABILITY TO GASES (CONSTANT VOLUME METHOD)

(Third Reprint June 2004)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 *November* 1976

AMENDMENT NO. 1 OCTOBER 1987

TO

IS:3400(Part 19)-1976 METHODS OF TEST FOR VULCANIZED RUBBERS

PART 19 PERMEABILITY TO GASES (CONSTANT VOLUME METHOD)

(Page 11, clause 11.1) - Substitute the following for the existing clause:

'11.1 The test report shall include the following particulars:

a) A reference to this standard;
b) The identification of the sample;
c) The gas used in the test;
d) The temperature of the test;
e) The thickness of the test piece; and
f) The permeability.'

(PCDC 13)

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Indian Standard

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PART XIX PERMEABILITY TO GASES (CONSTANT VOLUME METHOD)

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METHODS OF TEST FOR VULCANIZED RUBBERS

PART XIX PERMEABILITY TO GASES (CONSTANT VOLUME METHOD)

0. FOREWORD

0.1 This Indian Standard (Part XIX) was adopted by the Indian Standards Institution on 8 July 1976, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Chemical Division Council.

0.2 The measurement of permeability of rubber to gases is important in the evaluation of rubbers for such products as inner tubes, tubeless tyre liners, hoses, baloons or other gas containers, seals or membranes. The measurement is also of theoretical importance in the study of characteristics of diffusion and gas solubility in relation to polymer structure.

0.3 Because of dangers connected with high pressure and flammability, handling of gases should be done by experienced personnel only.

0.4 This standard is essentially based on ISO/R 1399-1970 'Determination of the permeability of vulcanized rubbers to gases (constant volume method)', published by International Organization for Standardization.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS:2-1960*.

1. SCOPE

1.1 This standard (Part XIX) prescribes the method for the determination of permeability to gases of vulcanized rubbers by constant volume method.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definition shall apply.

2.1 Permeability of Rubber to Gases — The rate of volume flow of gas under steady state conditions between opposite faces of a unit cube of solid rubber, for unit difference in pressure, when tested under controlled pressure and temperature.

*Rules for rounding off numerical values (*revised*).

3. OUTLINE OF THE METHOD

3.1 The cavity of a test cell, maintained at a constant temperature, is divided by a disc test piece into a high-pressure and a low-pressure (atmospheric) side. The gas permeates from high-pressure side into low-pressure side, which is of a very low dead volume and connected to a capillary tube; this is provided for measuring the permeated volume.

3.1.1 For normal comparison of permeability of different vulcanizates, the test temperature is the standard laboratory temperature ($27 \pm 1°C$), but higher preferred temperature may be used where conditions are required to approximate to the service temperature of rubber products.

4. APPARATUS

4.1 Test Cell

4.1.1 It consists of a test cell in which the test piece is clamped round its periphery in a gas-tight manner so as to expose one surface to gas under pressure. The other surface of the test piece shall be supported against the force due to the gas pressure so that no deformation takes place. For this reason the low-pressure side of the test cell shall be filled with a rigid, easily permeable, packing piece which may consist of a disc of microporous ebonite or discs of fine wire gauze which completely fill the cavity. A means of indicating gas pressure up to about 0·5 MPa (approx 5 kgf/cm^2), with an error of not more than 1 percent, shall be connected to the high-pressure side of the cell. Details of the apparatus are shown in Fig. 1 and 2.

4.1.2 The internal volume of the high-pressure side of the test cell shall be at least 25 cm^3 to minimize the pressure loss due to diffusion during the test which may last several hours.

4.1.3 The internal volume of the low-pressure (atmospheric) side of the test cell shall be kept to a minimum by the use of permeable packing as described above and by small diameter passages through the dismountable coupling and tubing to the manometer. For the design shown in Fig. 2, a total volume between test piece and datum mark of 1 to 2 cm^3 is typical. The test cell shall be of metal construction with sufficient mass to assist temperature stability, and should be provided with a drilled pocket to hold a suitable temperature measuring device.

4.2 Temperature Measuring Device — accurate to 0·2°C.

4.3 Volume Measuring Device — It consists fundamentally of a capillary tube of U shape of uniform cross section over the length for volume measurements. The capillary tube shall be graduated or a graduated scale shall be held close to it on the long straight portion.

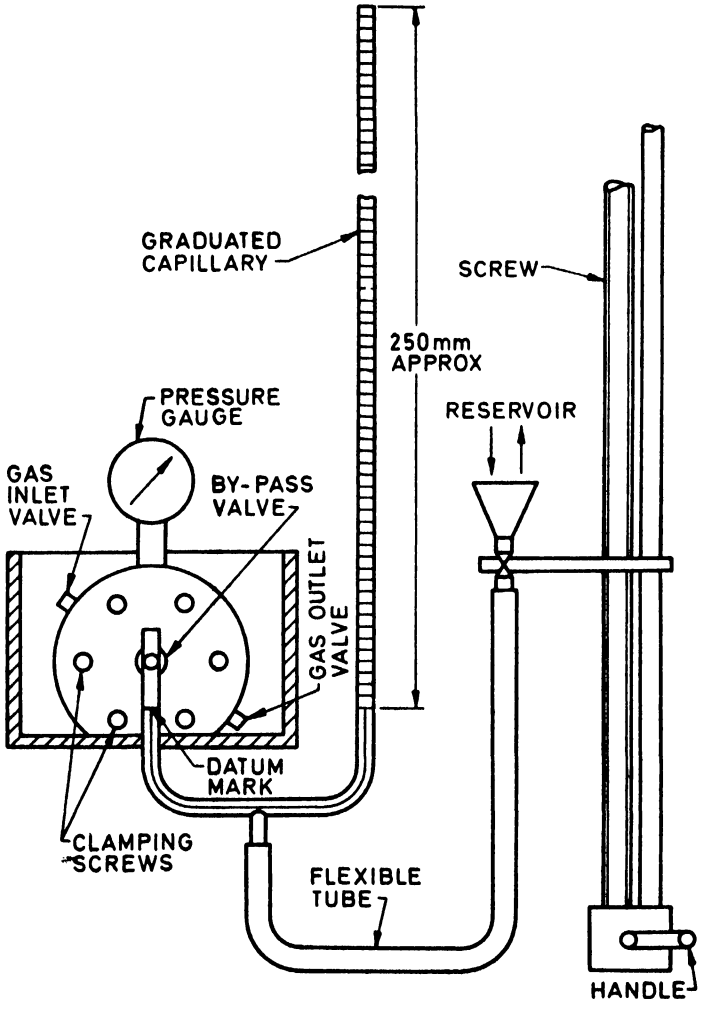

FIG. 1 PERMEABILITY APPARATUS

4.3.1 It shall be provided with a datum mark on the short portion close to the cell, and shall be suitably filled with a non-volatile liquid which does not dissolve the gas (suitable liquids are dioctyl sebacate or tritolyl phosphate, coloured with suitable colour). A microscope may be used to observe the liquid position.

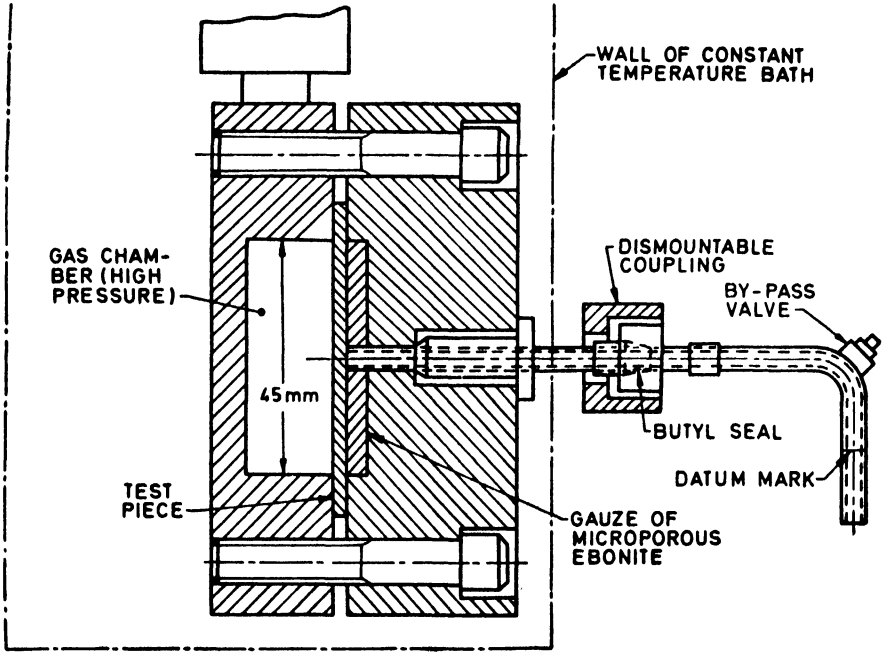

FIG. 2 SECTION SHOWING DETAILS OF DISMOUNTABLE COUPLING

4.3.2 A vertically adjustable reservoir of liquid shall be connected by a T-piece to the lowest manometer U-tube. A by-pass valve shall be provided between the union and datum mark, to release gas for initial adjustments.

4.4 Arrangement for Maintaining Constant Temperature — Constant temperature bath or other means capable of maintaining the test cell at the required test temperature to within $\pm 0{\cdot}5°C$. The wall of the bath shall be so arranged that the outlet from the test cell will project through the side, leaving the dismountable coupling accessible. A number of test cells containing different test pieces may then be connected in turn to a single manometer apparatus.

5. TEST PIECE

5.1 Shape and Dimensions — The test piece shall be a disc of uniform thickness and of dimensions to suit those of the test cell, and may be either moulded or cut from a test sheet or a product. It is preferable to use a moulded disc having on each face a circumferential rib or bead to fit into corresponding grooves in the clamping members. Where the test piece is a flat sheet, suitable separate O rings may be used to fit into the grooves.

The overall variation (excluding beads) in thickness shall not exceed 10 percent of the mean thickness. Suitable dimensions are 50 to 65 mm diameter with a free testing surface of 8 to 16 cm^2. The thickness may be between 0·25 and 3·0 mm, the smallest thickness being advantageous for rubber of low permeability such as isobutylene-isoprene rubber (IIR). There shall be no imperfections and pinholes.

5.2 Number of Test Pieces — Two test pieces from each rubber sample shall be tested.

6. TIME LAPSE BETWEEN VULCANIZATION AND TESTING

6.0 Unless otherwise specified for technical reasons the following requirements for time lapses shall be observed.

6.1 For all test purposes the minimum time between vulcanization and testing shall be 16 hours.

6.2 For non-product tests the maximum time between vulcanization and testing shall be 4 weeks and for evaluation intended to be comparable, the tests, as far as possible, should be carried out after the same time interval.

6.3 For product tests, whenever possible, the time between vulcanization and testing shall not exceed 3 months. In other cases tests shall be made within 2 months of the date of receipt by the purchaser of the product.

7. TEMPERATURE OF TEST

7.1 For normal comparisons of permeability of different rubber vulcanizates, the test temperature shall be standard laboratory temperature, that is $27 \pm 1°C$. Higher temperature may be used where conditions are required to approximate to service temperature of rubber products. Such higher temperature and their tolerances shall be selected as agreed by the purchaser and the supplier.

8. PROCEDURE

8.1 Preparation of Test Piece

8.1.1 Examine the test piece carefully for pinholes or imperfections within the area of the internal diameter of the cell (which is the effective test area) and free the test piece from all surface contaminations, such as wax or film of mould lubricant by means of organic solvent which neither attack nor swell the rubber.

8.1.2 Determine the thickness of the test piece in the test area as the average of six measurements each made to an accuracy of 0·02 mm. Insert the permeable packing in the shallow cavity in contact with the test piece. Clamp the test piece securely round its periphery.

NOTE — It is permitted to use a small amount of vacuum grease on the clamping surfaces to secure gas-tightness. No grease shall be allowed to appear on the central area of the test piece. With flat test pieces (that is without beads) of thickness 0·5 mm or less, washers of soft vulcanized isobutylene-isoprene rubber (IIR) on both sides of the test piece, may be necessary to ensure a gas-tight seal. Fill the gas chamber (*see* Fig. 2) with the test gas to the required pressure of test, usually 0·2 to 0·4 MPa (approx 2 to 4 kgf/cm²). Then bring the test cell to the test temperature, and couple the manometer tube by means of the union.

8.2 Conditioning of Test Piece — The assembled apparatus shall be allowed to remain at the test temperature for a minimum of 24 hours, or, where the approximate value of the diffusivity is known, for a minimum time t derived from the following equation:

$$t = \frac{b^2}{2Q} \times S = \frac{b^2}{2D}$$

where

t = the conditioning time in seconds,

b = the thickness in metres of the test piece,

Q = the permeability coefficient in $\frac{m^2}{Pa.s}$,

S = the gas volume in cubic centimetres absorbed by 1 cm^3 of the test piece at a pressure of 1 Pa (approx 1×10^{-5} kgf/cm^2), and

D = the diffusion coefficient in square metre per second.

The minimum time t ensures that the diffusion of gas through the test piece, and hence the gas flow through the test piece, may reach the steady state corresponding to the right-hand (straight) portion of the curve in Fig. 3. The left-hand portion of this curve indicates the initial approach to steady conditions due to diffusion through the test piece. The strictly linear portion of the curve only is used for permeability measurement.

8.3 Determination of Permeability to Gas — Fill the capillary tube with the non-volatile liquid specified in **4.3.1**. With the by-pass valve open to atmosphere, adjust the liquid reservoir to bring the liquid level above the datum mark. If a gas other than air is used, the gas chamber of the test cell shall be flushed out with the test gas prior to commencement of test. Then close the by-pass valve. As the gas diffuses through the test piece, the meniscus descends; when it crosses the datum mark, start the stop-watch (that is zero time). Raise the reservoir again to bring the meniscus above the datum mark, and note the time and manometer reading (gas volume) when the meniscus again passes the mark. Repeat this procedure until sufficient readings have been obtained to establish with adequate

accuracy the slope of the straight part of the time/manometer reading (gas volume) curve (*see* Fig. 3). If the time/manometer reading curve shows any appreciable departure from linearity in the sense of curvating towards the right, this shows that there is leakage from the low-pressure side of the apparatus. If this occurs, the test result shall be rejected, and the apparatus dismantled and re-assembled.

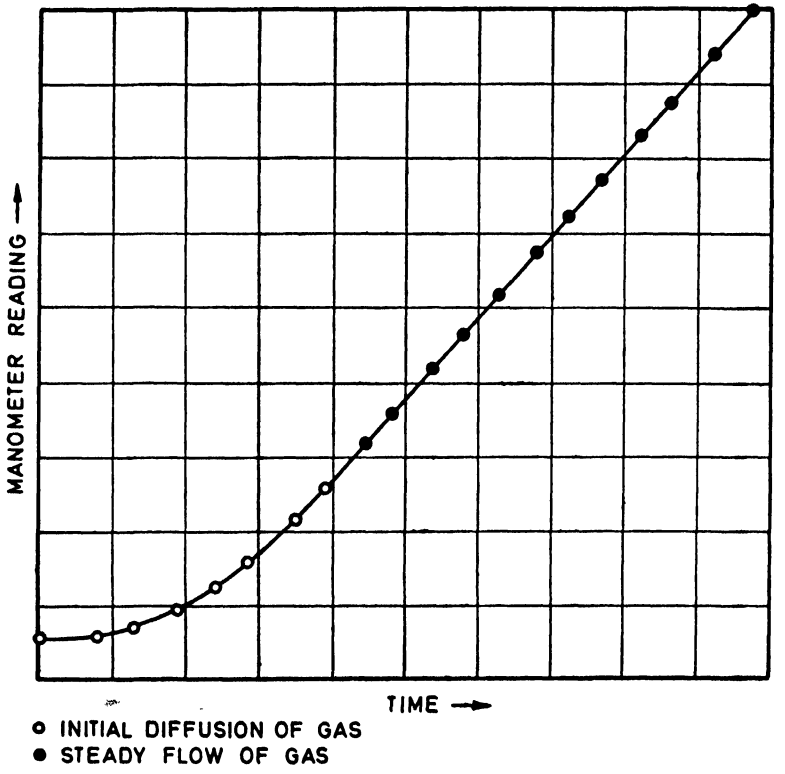

FIG. 3 TYPICAL TIME/MANOMETER READING CURVE

8.4 Duration of Test — The duration of test of a single test piece shall usually be of the order of 15 to 30 minutes, readings being taken about every 2 minutes and plotted on a graph, such as shown in Fig. 3, to check that steady conditions have been reached.

9. EXPRESSION OF RESULTS

9.1 The measurement of rate of gas flow shall be carried out by noting the rate of increase of pressure at constant volume. It is, therefore, important

to calibrate the apparatus by an accurate determination of the gas volume between the test piece and the datum mark, by measuring the dimensions of the cavity and adding the volume of the passages and tubing.

9.2 The effective volume of the permeable packing which is inserted in the test cell may be calculated by dividing the mass of the packing by the density of the material from which it is made; for example, if copper gauze is used for the packing material the total mass of the copper gauze shall be divided by the density of solid copper, that is 8·8 g/cm³. The effective volume of the packing shall then be deducted from the internal volume of the low-pressure side of the apparatus up to the datum mark.

The SI unit in which permeability measured is $\frac{m^2}{Pa.S}$ and the typical figures for a natural rubber gum stock are of the order of 9×10^{-17} to 15×10^{-17} units.

The permeability in $\frac{m^2}{Pa.S}$ shall be calculated from the following formula:

$$\frac{dh}{dt} \times \frac{v_0 b\rho \times 273 \times 10^3 \times 9{\cdot}81}{A \times P \times T \times 10^5}$$

$$\text{or} \quad \frac{dh}{dt} \times \frac{v_0 bP}{APT} \times 267\,813$$

where

$\frac{dh}{dt}$ = the manometer rise in metres per second,

v_0 = the effective gas volume in cubic metres on the low-pressure side of the test cell,

b = the thickness in metres of the test piece,

ρ = the density in g/cm³ of the manometer liquid,

A = the area in square metres of the test piece (neglecting the clamped area),

P = the pressure difference in pascal of the diffusing gas across the test piece,

T = the temperature in kelvin, and

10^5 = approximately normal atmospheric pressure in pascal.

10. REPRODUCIBILITY OF RESULTS

10.1 The accuracy of test by the method described shall usually be of the order of ± 5 percent coefficient of variation for repeat tests on a single test piece. The coefficient of variation between repeat vulcanizates of the same compounding formula may give rise to a further error of ± 3 percent.

11. TEST REPORT

11.1 The test report shall include the following particulars:

a) the permeability,

b) the gas used in the test,

c) the temperature of the test, and

d) the thickness of the test piece.

(*Continued from page* 2)

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